

## Measurement of Detection Efficiency in Atom Probe Tomography

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Atom Probe Tomography (APT) is a near-atomic-scale analytical technique, and due to recent advances in instrumentation and sample preparation techniques, APT is being used on a variety of increasingly challenging 3D applications [1]. Total system detection efficiency is a key parameter for 1) estimating system performance, 2) understanding the detection of low concentration species and small clusters of atoms, and 3) accurate reconstruction of atomic coordinates from detected ions; however, its experimental determination can be complicated. The techniques used to date provide limited precision and accuracy because alternative means have not been reported [2,3]. These methods utilize the atomic periodicities observed in data and require significant data manipulation where each step may be prone to biasing the end result.

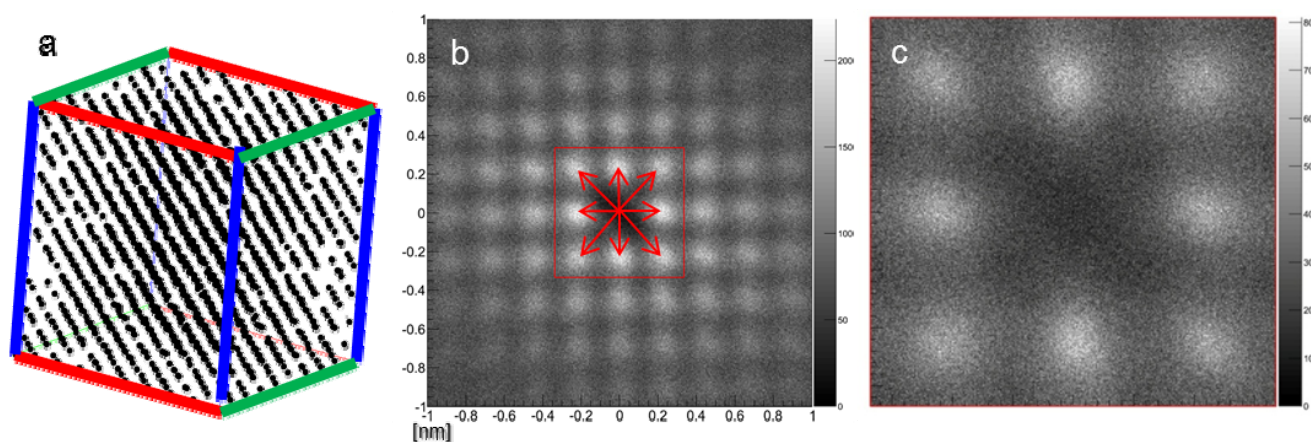
With the goal of extracting reliable efficiency measurements in mind, we explored two different approaches for measuring efficiency. The first is an extension of the lattice occupancy approach mentioned above, while the second considers measured atom counts from features of known volume. The ability to compare the results from two different approaches provides a basis for estimating the accuracy of each method.

The lattice occupancy method studied here is based on analysis of reconstructed crystal volumes with a spatial distribution map (SDM) and is illustrated in Fig. 1 [2]. First, a reconstruction must be properly calibrated so that the experimental periodicity is aligned to the known crystallography. Next, an SDM histogram is collected to measure occupancy rates of nearby lattice sites. Only data sub-volumes of uniform density and periodicity are considered to achieve reasonable measurement precision. Efficiency is then calculated by comparing the average relative lattice occupancy rates observed to those expected. Total detection efficiencies of 59% have been measured for the LEAP 4000Si.

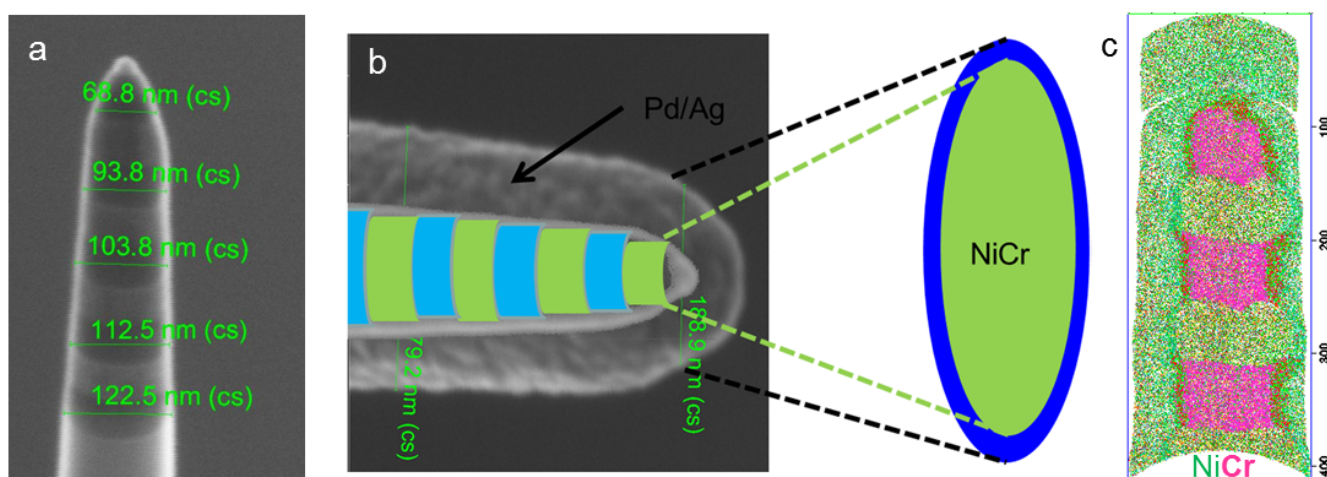
We also report on an alternative method for measuring total system detection efficiency as well as the necessary specimen characteristics required to support the method. As illustrated in Fig. 2, APT analysis of known well defined volumes of material coupled with a high quality measurement of the volume dimensions allows for estimation of the number of atoms within the volume. Adequately encapsulating these volumes ensures that all the field evaporated ions are incident on the detector. The simple ratio of the detected number of ions to the expected number of atoms then provides a direct measurement of efficiency. We present efficiency measurements based on NIST SRM 2135c encapsulated in silver and nickel as well as progress on alternate materials and encapsulations.

### References:

- [1] T.F. Kelly and D.J. Larson, *Ann. Rev. Mat. Res.* **42** (2012), p. 1.
- [2] B.P. Geiser *et al*, *Microsc. Microanal.* **13** (2007), p. 437.
- [3] B. Deconihout, *et al*, *Rev. Sci. Inst.* **73** (2002), p. 1734.



**Figure 1.** a) A 3D volume of tungsten atoms is converted into a 3D SDM histogram. b) The central slice with the [110] plane is extracted and c) the 8 lattice sites around the SDM center are considered for an efficiency measurement. The ratio of the total observed nearest neighbors to the expected number of atoms in those 8 lattice sites provides a measure of the system detection efficiency.



**Figure 2.** a) An SEM image of an atom probe specimen composed of alternating nickel and chromium layers shows the measurements of each layer diameter. b) The specimen is encapsulated in sufficient sacrificial material (in this image a palladium seed followed by silver) so that the field evaporated multi-layers will reside within the field-of-view of the detector and only a portion of the encapsulant will not be detectable. c) A reconstructed ion map shows the original nickel (green) chromium (magenta) multi-layers as well as some of the encapsulating layer (in this case nickel). The ratio of the measured atoms to those expected based on the measured volumes provides a measure of the system detection efficiency.